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# SPECIFICATION FOR CHINA CLAY FOR CERAMIC INDUSTRY

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## SPECIFICATION FOR CHINA CLAY FOR CERAMIC INDUSTRY

C. S. 161: 1972

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## CEYLON STANDARD SPECIFICATION FOR CHINA CLAY FOR CERAMIC INDUSTRY

#### **FOREWORD**

This Ceylon Standard has been prepared by the Drafting Committee on Ceramics. It was approved by the Civil Engineering Divisional Committee of the Bureau of Ceylon Standards, and was authorised for adoption and publication by the Council of the Bureau on 3rd October, 1972.

China Clay or Kaolin is the name given to clays which form the basis of high quality domestic pottery and which nowadays are known to consist mainly of the clay mineral kaolinite (A1203·2 Si02 2H20). Kaolin is the name derived from Chinese Kaolin, a high ridge where the white firing clay was first discovered.

Raw China clay is usually refined to obtain a Kaolin of high purity suitable for industrial purposes. China clay on the whole contains less fine material than ball clay. Consequently, they are less plastic, and have less binding power than ball clay.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated expressing the result of a test or observation shall be rounded off in accordance with C. S. 102: Ceylon Standard on Presentation of Numerical Values. The number of figures to be retained in the rounded off values shall be the same as that of the specified value in this standard.

In the preparation of this standard, the considerable assistance obtained from the Department of Geological Survey, Ceylon Ceramics Corporation and the publication of the ISI is acknowledged.

#### 1 SCOPE

This standard prescribes the requirements and the mothods of sampling and test for China clay used in the ceramic industry.

## 2. REQUIREMENTS

2.1 The material shall be the natural mineral consisting essentially of hydrated aluminium silicate, suitably beneficiated to remove extraneous substances.

#### C. S. 161 · 1972

- 2.2 The material shall also comply with the requirements laid down in Table 1, when tested in accordance with the methods prescribed in Appendices 'A' and 'B'
- 2.3 The moisture content of the material shall be as agreed to between the purchaser and the supplier.
  - 2.3.1 Where moisture content is specified by the purchaser, it shall be determined by the method specified in Appendix 'C'.

#### 3. SAMPLING

Representative samples of the material shall be drawn as prescribed below:

TABLE 1
REQUIREMENTS FOR CHINA CLAY FOR
CERAMIC INDUSTRY

Serial No.	Characteristic	Require- ments	Method of Test
1.	Percentage remaining on 53µm sieve (max.)	1	A-1
$\frac{1}{2}$ .	Deposited a reging through 0	1	
Δ'	Percentage passing through 2 µm sieve (min.)	<b>7</b> 0	A-2
$3 \cdot$	Loss on ignition,% by mass (min)	12	A-3
4.	Water of Plasticity (min.)	25	A-4
5 ·	Dry (linear) shrinkage at 110°C (max)	10	A-5
$6 \cdot$	Total fired shrinkage at S. K 12/6a-1200°C		11.0
	(max.)	18	A-6
7 ·	Alumina as Al <sub>2</sub> 0 <sub>3</sub> ,% by mass (min.)	37	В
8.	Oxides of iron (as Fe <sub>2</sub> 0 <sub>3</sub> ),% by mass (max.)	1.00	B
9.	Titanium dioxide (TiO <sub>2</sub> ).% by mass (max.)	0.80	B
10 ·	Titanium dioxide and feric oxide together	0 00	ע
	(Fe <sub>2</sub> O <sub>3</sub> +TiO <sub>2</sub> ) (max.)% by mass	1.5	_

## 3.1 General requirements of sampling

- 3.1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.
- 3.1.2 Samples shall not be taken in an exposed place.
- 3.1.3 Sampling instrument shall be clean and dry when used.

- 3.1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.
- 3.1.5 The samples shall be placed in clean dry air tight glass or other suitable containers.
- 3.1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.
- 3.1.7 Each sample container shall be sealed air tight with a stopper after filling and marked with full details of sample, the date of sampling and other identification particulars.
- 3.1.8 The samples shall be stored in such a manner that during storage the properties of the material do not get affected.
- 3.1.9 The sampling shall be done by a person or persons mutually agreed to between the purchaser and the supplier.

#### 3.2 Scale of sampling for material packed in bags or other containers

- 3.2.1 Lot In a single consignment all the material belonging to the same grade and coming from the same supplier or source shall constitute a lot.
- 3.2.2 For the purpose of sampling, all the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal mass in accordance with col. 1 and 2 of Table 2. These containers shall be selected at random from the sub-lot. From each of these containers a small but equal increment of material shall be taken.

TABLE 2
SCALE OF SAMPLING FOR CONTAINERS

No. of containers in the lot	No. of sub-lots	No. of bags to be sampled from each sub-lot
(1)	(2)	(3)
Up to 10 11 to 200 201 to 500 501 to 2 000 2 001 and above	1 2 3 4 5	AlI 5 10 15 220

All the increments from the sample containers in the same sub-lot shall be pooled together to constitute a gross sample so that there are as many gross samples as the sub-lots into which a lot has been divided.

## 3.3 Preparation of samples

- 3.3.1 All the increments from the same sub-lot shall be thoroughly mixed to constitute a gross sample representing the sub-lot. The minimum size of the gross sample shall be about 3kg. If the gross sample is less than this, additional number of increments shall be drawn so as to make up the required quantity.
- 3·3·2 The gross sample shall be suitably reduced by the method of coning and quartering or any other suitable method to about 750g which shall be divided into three portions called laboratory samples, one for the purchaser, another for the supplier and the third as the referee sample each duly placed in the sample container.

#### 4. PACKING

The material shall be packed in 3-ply paper bags or as agreed to between the purchaser and the supplier.

#### 5. MARKING

The bags shall be securely closed and marked indelibly with the following:-

- (1) Name of the manufacturer;
- (2) Nett mass;
- (3) Batch/Code Number.

## APPENDIX 'A'

## PHYSICAL ANALYSIS OF CHINA CLAY

## A-1 DETERMINATION OF FINENESS

## A-1-1 Reagent

A-1·1·1 Ethyl alcohol 95 per cent (v/v)

- A-1.2 Procedure Place 10.0g of the material previously dried to constant weight at 100° ± 5°C in a tared 53 µm C.S. Sieve. Wash by means of a slow stream of running water and then with a fine jet of water from a wash bottle until all the material that can pass through the sieve has been washed. Wash finally with a small quantity (about 10ml) of ethyl alcohol and then air dry to constant mass.
- A-1-3 Report the material remaining on the sieve as the percentage of the air dried material taken.

## A - 2 DETERMINATION OF PARTICLE SIZE DISTRIBUTION

### A-2.0 Principle

A-2.0.1 The progress of sedimentation of a clay suspension in a tall cylinder is followed by withdrawing sample portions at a known depth based on the temperature of the suspension below the surface, with a picette and weighing the sediment. Stokes' law is then applied to find out the grain size.

### A-2.1 Apparatus

A-2.1.1 The Andreasen Pipette - The pipette - (see Fig. 1) consists of a graduated cylinder (W) not less than 60mm in diameter and having a capacity of 550ml when filed to the upper mark on the scale. It is provided with a ground glass stopper through which passes the stem of a pipette which extends 200mm beneath the surface of the suspension and about 40mm from the bottom, the tip being at the level of the zero mark on the scale while the original surface

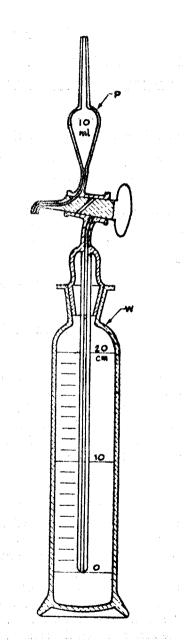


FIG. I - Andreasen Pipette

of the suspension is at the 200mm mark. The one mark-pipette (P) has a capacity of 10ml, and is provided with a three-way stopcock just obove the ground glass joint. The stopcock regulates flow in the tube inside the cylinder, the pipette (P) and a draining tube for drawing the clay suspension in a weighed porcelain basin or silica dish.

A-2.1.2 Basin - Porcelain, or silica dish.

#### A-2.2 Reagents

A-2.2.1 Sodium pyrophosphate solution - Dissolve 24 55g of sodium pyrophosphate (Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>·10H<sub>2</sub>O) in 500ml of water.

#### A-2-3 Procedure

- A-2.3.1 Preparation of sample Crush the clay sample to pass through a 4.75mm C.S. Sieve. Determine the moisture by drying a portion of the sample at 110°C.
- A-2·3·2 Place 5·50g of clay sample (calculated, dry basis) in a 250ml wide mouth bottle and add 200ml of distilled water. Allow to stand for 24 hours. Add 10ml of the sodium pyrophosphate solution. Agitate the bottle with its contents by rotating end over-end for 17 hours
  - Note: This is best done by packing the bottle in the jar of a ball mill so as to turn end over-end as the mill rotates.
- A-2·3·3 Transfer the dispersed sample to the Andreasen pipette and add distilled water to bring the level of the suspension to the upper mark on the scale, making the total volume 550ml.
  - Note: This produces a 1 percent suspension, and the concentration of the electrolyte is 0.002 Mol/1 (gram molecule per litre).
- A-2-3-4 Place the Andreasen pipette in a constant temperature bath at a temperature of 30°C, for 15 to 30 minutes to permit it to attain the temperature of the bath.

Remove it, shake by hand and replace immediately in the bath. Withdraw 10ml of suspension at increasing intervals of time, at a uniform rate of about 20 seconds. The samples are drawn at the interval of 3 minutes and 30 minutes, and then at 1, 2, 3, 5, 8, 24 and 96 hours.

Note: These intervals are chosen arbitrarily, but are adhered to in order to save time in calculating the grain size.

A-2·3·5 Take 10ml aliquots in small, weighed porcelain basins, and evaporate to dryness at 110°C. Weigh the residue accurately to 0·1mg.

#### A-2.4 Calculations

A-2.4.1 Calculate the percentage of the dried residue as follows:

Mass of dried residue.

M percent =  $\frac{M_1}{M_2} \times 100$ 

where,

M<sub>1</sub> = mass of the dried residue, and

 $M_2$  = mass of the clay sample.

Note: Make a correction for the sodium pyrophosphate percent in the mass of the dried residue,  $M_1$ .

A-2.4.2 Calculation of size distribution - The grain sizes are calculated from Stokes' law which may be expressed as follows:-

$$r = \sqrt{\frac{g h \eta}{2 (D_1 - D_2) gt}}$$

where.

r = radius of spherical particle in mm.

h = distance in mm between liquid surface and pipette tip when suspension is drawn.

 $\eta$  = viscosity of suspending medium (g/cm sec)

D<sub>1</sub> = Density of the particle

D<sub>2</sub> = Density of suspending medium

g = gravitation constant  $(9.81 \text{ m/s}^2)$  and,

t = time in seconds from start of the test.

(a) When the test is conducted at 30°C, the clay having a density of 2.65 and distilled water as the suspending medium, the above formula may be expressed as follows:

$$r = 1.923 \sqrt{\frac{h}{t}}$$

where r is expressed in µm.

(b) Stokes' law is applicable to angular or cubical particles of the same weight as spherical particles. By calculating the grain size as the edge length, or width, of a cube of the same volume as a sphere of radius r, the width of the cube may be expressed as follows:

Width of the cube, C = 1.612 r

Note: This width of the cube shall be adopted for expressing the results of test.

- (c) Plot a smooth graph with (C) as abscissae and the percentage by mass (m) finer then the corresponding size as ordinate. From the curve find out the particle size distribution for the specified sizes.
- (d) Duplicate tests shall be made on each sample. If the mass percentages vary more than one percent, the test shall be repeated.

## A-3 DETERMINATION OF LOSS ON IGNITION

- A-3.1 Take about 50g of the material in a China dish and heat to constant mass at 105° + 2°C. Cool it in a desiccator and keep for subsequent tests
- A-3 2 Heat 1g of the above dried material in a platinum crucible for 15 minutes over a small flame, then for 30 minutes over a good Meker burner, with the lid on for the last 10 minutes. Cool the crucible and contents in a desiccator, weigh, again heat for 10 minutes over the Meker burner and then weigh again after cooling. The loss in weight denotes loss in ignition and the percentage of loss may be calculated.

#### A-4 DETERMINATION OF WATER OF PLASTICITY

A-4.1 Principle - The water content of the clay at the point of maximum workability is determined. This when expressed as a percentage on oven dried clay, gives the value for water of plasticity.

#### A-4-2 Apparatus

- A-4.2.1 Spatula
- A-4.2.2 Porcelain Basin
- A-4.2.3 Burette 50 ml capacity
- A-4.2.4 Balance accurate up to 0.01g
- A-4 · 2 · 5 250µm C.S Sieve

#### A-4-3 Procedure

- A 4·3·1 Dry thoroughly about 500g of clay at a temperature of 70°C and crush to pass 250μm C.S. Sieve. Work up the sieved clay with water, by adding small quantities of water from the burette, to a soft plastic consistency and thoroughly wedge and knead by hand. Allow it to age for 24 hours keeping it covered by wet cloth during the period. After 24 hours, knead the plastic mass again and pug it well by further small additions of water until proper working consistency for either extrusion or moulding into bars is obtained. Take three equal portions of the clay mass, at this consistency from different portions, the edges and corners rounded to avoid any handling loss, and weigh immediately to the nearest 0.01g.
- A-4·3·2 Dry the three portions of the clay mass at 110 ± 5°C to constant weight. Cool them in a desiccator and weigh to the nearest 0·01g.

#### A 4.4 Calculation

Water of plasticity, percent by mass  $=\frac{M-M_1}{M_1} \times 100$ 

Where,

M = mass in g of the plastic clay mass, and

M<sub>1</sub> = mass in g of the dried clay mass.

Note: Round off the calculated value to the second place of decimal.

A-4.5 Report - Report the average of the three specimens.

## A 5 DETERMINATION OF DRY LINEAR SHRINKAGE

A 5 I Principle The dry shrinkage of a clay is determined by measuring the decrease in one dimension of a clay mass, when the water content is reduced from the plastic state to the dry state at 110°C.

#### A-5.2 Apparatus

- A-5.2.1 Mould A collapsible wooden or other suitable mould with internal dimension 25 x 25 x 120mm suitable for forming the test specimen of that size.
- A-5.2.2 Micrometer Graduated to at least 0.02mm, to an accuracy of ± 0.05mm.

#### A-5.3 Procedure

A-5·3·1 Take a portion of clay as prepared in A-4·3·1 and again knead thoroughly. Coat evenly the inside of the mould with a thin layer of kerosene oil or light machine oil. Press a lump of the plastic clay mass into the mould. Take care to avoid entrapping any air. Strip off any excess clay with a straight edged spatula from the top of the mould and finally wipe off any adhering clay. Make the surface of clay smooth and mark two reference points approximately 100mm apart. Measure the distance between the two points accurately to second place of decimal with the micrometer. Prepare five such test specimens.

A-5·3·2 Carefully release the test specimens from the moulds and allow to dry slowly under a wet cloth for 48 hours. Air dry the specimen for another 48 hours at room temperature, occasionally turning sides of the test specimens to expose new surfaces for evaporation. Place the test specimens in an air oven, preferably with automatic temperature controls, and dry them at 110°±5 C for 24 hours, to constant mass. Remove the test specimens from the oven and keep in a desiccator to cool. Measure accurately to second place of decimal the distance between the two reference points.

#### A-5.4 Calculation

A-5.4.1 Calculate the linear dry shrinkage as follows:

Linear dry shrinkage, percentage =  $\frac{L_p - L_d}{L_d} \times 100$  Where,

Lp = length of the plastic clay mass in mm, and

 $L_d$  = length of the dry clay mass in mm

Note: Round off the calculated value to the nearest second place of decimal.

A-5.4.2 Report the average of the five test specimens.

## A-6 DETERMINATION OF FIRED LINEAR SHRINKAGE

- A-6.1 Principle The decrease in one dimension of a dry clay mass heated to the required temperature is measured.
- A-6.2 Procedure Take five test specimens as prepared in A-5.3.2. Place them in a suitable furnace, with Segar cone 12/6a-1200 and heat it till the cone bends over and its tip touches the plaque. Remove the test specimens and cool in a desiccator. Measure the distance between the two reference points with a micrometer accurately to the second place of decimal.

#### A-6.3 Calculation

A-6.3.1 Calculate the linear fired shrinkage as follows:

Linear fired shrinkage, percentage =  $\frac{L_d - L_f}{L_i} \times 100$ 

where,

L<sub>d</sub> = length of the dry test specimen in mm, and

 $L_f$  = length of the fired test specimen in mm.

Note: Round off to the nearest second place of decimal.

A 6.3.2 Report the average of five test specimens.

## APPENDIX 'B'

## CHEMICAL ANALYSIS OF CHINA CLAY B-0 QUALITY OF REAGENTS

- B-0·1 Unless otherwise specified, pure chemicals and distilled water shall be used.
  - Note:- 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## B-1 DETERMINATION OF OXIDES OF IRON, TITANIUM AND ALUMINIUM

B-1·1 Principle - China clay is decomposed by sintering with fusion mixture and the fired mass is extracted with hydrochloric acid. Silica is dehydrated by baking and is filtered. From the filtrate hydroxides of iron, titanium and aluminium are precipitated by ammonia, filtered washed and ignited. From the ignited mixed oxide ferric oxide and titanium dioxide are estimated and alumina is found out by difference.

## B-1.2 Reagents

- B-1·2·1 Fusion mixture Mix the carbonates of sodium and potassium in equal proportions.
- **B-1.2.2** Dilute hydrochloric acid -1:1 and 1:4 (by volume)
- B-1.2.3 Concentrated sulphuric acid sp. gr. 1.84
- B-1.2.4 Hydrofluoric acid 40 percent.
- B-1.2.5 Potassium bisulphate solid

- B-1.2.6 Ammonium chloride solid
- B-1.2.7 Dilute nitric acid 1:1 (by volume)
- B-1.2.8 Methyl red indicator solution Dissolve 0.15g of methyl red in 500ml of distilled water.
- B-1.2.9 Concentrated ammonium hydroxide sp. gr. 0.90
- B-1.2.10 Ammoniacal ammonium nitrate solution Dissolve one gram of ammonium nitrate in 100ml of water and make it slightly alkaline to methyl red with dilute ammonia.

#### B-1.3 Procedure

- B-1.3.1 Mix thoroughly about 0.5g of the accurately weighed test sample with 5g of fusion mixture in a covered platinum crucible and cover the mixture with a layer of the fusion mixture. Raise the temperature gradually until frothing ceases and then complete the fusion by maintaining the temperature at 900° to 950°C for about 30 minutes.
- B-1.3.2 Cool, place the crucible and lid in a porcelain basin and add 30ml of dilute hydrochloric acid (1:1). Warm until the melt has disintegrated, wash the crucible and lid with dilute hydrochloric acid (1:4), taking care to remove all silica Break all lumps with a glass rod and evaporate slowly to dryness over a steam bath or on an electric hot plate breaking occasionally the crust that may hinder the evaporation. Bake for an hour at 110°C, preferably on a sand bath to drive off hydrochloric acid. Add 20 to 30ml of dilute hydrochloric acid (1:1) and 50ml of hot water and heat to boiling to dissolve the soluble salts. Allow to settle for a few minutes and then filter through filter paper (Whatman No. 41 or equivalent is suitable). Wash the silica first with dilute hydrochloric acid (1:4) and then with hot water until completely free from chloride. Preserve the paper and the residue (A).

- B-1·3·3 Transfer the filtrate back to the dish. Again evaporate the filtrate and re bake for dehydration of silica. Moisten the residue with a few drops of dilute hydrochloric acid solution (1:1) and hot water, filter, wash and collect the silica residue (B) as under B-1·3·2. Preserve the filtrate for the determination of mixed oxides.
- B-1.3.4 Place silica residues (A) and (B) without drying in a platinum crucible and heat cautiously to dry the residue and char the papers. Burn off the carbon and ignite at 1 100°C. Cool. Moisten the contents with 1ml of water, add three to four drops of concentrated sulphuric acid and 15ml of hydrofluoric acid. Evaporate the solution to dryness on a sand bath or under an infra red lamp, heat again slowly at first, and then on the Blast Meker for 1-2 minutes. Preserve the residue (C) for determination of mixed oxides.
- B-1·3·5 Fuse the residue (C) preserved under B-1·3·4 in the crucible, with one gram of potassium bisulphate, cool and extract with a small amount of water acidulated with concentrated sulphuric acid. Add to the filtrate reserved under B-1·3·3. Add about 5g of ammonium chloride and a few drops of dilute nitric acid and heat almost to boiling. Add three drops of methyl red indicator and add slowly 1:1 ammonium hydroxide until the colour of the solution changes to yellow. Boil the alkaline solution for one to two minutes to remove the excess ammonia; allow to settle for about 5 minutes and filter through filter paper (Whatman No. 41 or equivalent is suitable).
- B-1·3·6 Puncture the bottom of the filter paper, wash down the precipitate completely into the original beaker first with water and then with 10ml of concentrated hydrochloric acid and 50ml of hot water. Place the filter paper inside the beaker and macerate well. Place the covered beaker on the steam bath for about 15 minutes

for complete solution of the hydroxides. Dilute to 200ml, precipitate and filter as described under B-1 3.5 and wash with hot ammoniacal ammonium nitrate.

B-1.3.7 Wash the precipitate thoroughly till it is free from chloride, place it in a weighed platinum crucible burn filter paper slowly and ignite at 1 000°C to constant mass. This ignited residue of mixed oxides consists of ferric oxide, titania and alumina. Percentage of mixed oxides =  $\frac{\text{Difference in mass of platinum crucible}}{\text{mass of sample}} \times 100$ 

## B-2 DETERMINATION OF FERRIC OXIDE

B-2·1 Principle – The ignited residue of mixed oxides is fused with an alkaline bisulphate and the fused mass extracted with sulphuric acid. After removal of silica, iron in the filtrate is determined by the colour developed with 1:10 orthophenanthroline and the optical density of the solution is measured with the help of an absorptiometer and compared with the standard.

## B-2.2 Reagents

- B-2.2.1 Potassium bisulphate solid
- B-2.2.2 Sodium bisulphate solid
  - B-2.2.3 Dilute sulphuric acid 1:9 (by volume)
  - B-2.2.4 1:10 Orthophenanthroline 0.1% solution
  - B-2.2.5 Hydroxylamine hydrochloride 10% solution
  - B-2.2.6 Sodium citrate 10% solution
  - B-2.2.7 Standard iron solution (1ml = 0.001g of Fe<sub>2</sub> O<sub>3</sub>).

    Dissolve 0.4911g of ferrous ammonium sulphate in water and add 10ml of dilute sulphuric acid (1:1) and 5ml of hydrogen peroxide (20%). Boil for 15 minutes to decompose the excess of hydrogen peroxide. Cool and dilute to 1,000ml in a measuring flask.

#### B-2.3 Procedure

- **B-2·3·1** Fuse the residue obtained under B-1·3·7 with potassium or sodium bisulphate in a porcelain crucible. Dissolve the melt in dilute sulphuric acid (1:9) and evaporate to fumes. Cool, dilute with water and filter off any silica. Wash the residue with hot water. Make up the volume of the filtrate to a standard (say 100ml) with dilute sulphuric acid.
- B-2.3.2 To a set of three 100ml volumetric flasks, add nothing to the first (to serve as a blank). Pipette suitable aliquots of the standard and sample solution to the other two, depending on the amount of iron present in the sample solution. Add 5ml of hydroxylamine hydrochloride solution to each flask with a graduated cylinder and allow to stand for 10 minutes. Add 10ml of orthophenanthroline solution followed by 10ml of sodium citrate solution and stir well. Dilute to the mark with distilled water, mix and keep for one hour. Measure the optical density at 560m µ (or appropriate filter) of the sample and the standard solutions. using reagent blank as the reference blank solution. Calculate the concentration or iron in the sample solution by comparing with the standard and hence the percentage of iron in the original solution.

## **B-3 DETERMINATION OF TITANIA**

B-3.1 Outline of the method – To the sulphuric acid solution of the ignited mixed oxides hydrogen peroxide is added and the colour due to iron is suppressed by means of phosphoric acid. The titanium in the solution is determined colorimetrically with the help of an absorptiometer and compared with the standard.

## B-3.2 Reagents

- $B-3\cdot2\cdot1$  Dilute sulphuric acid -1:20 (by volume)
- B-3.2.2 Phosphoric acid 85%
- B-3.2.3 Hydrogen peroxide 30%

- B-3.2.4 Standard titanium solution (1ml = 0.001g of TiO<sub>2</sub>). Weigh exactly 0.500g of pure calcined titania previously dried at 110°C. Fuse with 8.10g of potassium bisulphate in a clean platinum crucible, keep at a temperature as low as possible to maintain fluidity. Cool the fused mass and dissolve in about 300ml of dilute sulphuric acid filter and transfer to a 500ml graduated flask and make up to the mark with dilute sulphuric acid (1:20), mix thoroughly. Take 10ml of this solution in a 100ml graduated flask and dilute with water to the mark.
- B-3.3 Procedure To a set of three 100ml volumetric flasks, add nothing to the first (to serve as a blank). Pipette suitable aliquots of the sample and standard solutions to the other two flasks, depending on the titanium present in the sample solution. Add 50ml of dilute sulphuric acid to each of the flasks with a graduated cylinder and sufficient phosphoric acid to the flask containing sample solution to decolourise the iron. Add the same amount of phosphoric acid to the other two flasks. Add 5ml of hydrogen peroxide solution to all three flasks, mix well, dilute to the mark with distilled water. Mix, keep for half an hour and measure the optical density of the sample and the standard solutions at 410m \(\mu\). (or appropriate filter) using reagent blank as the reference blank solution. Calculate the concentration of titanium in the sample solution by comparing with the standard and hence the amount of titania in the original solution.

### **B-4 DETERMINATION ALUMINA**

B-4.1 Principle - Alumina is estimated by difference.

B-4.2 Calculation

Alumina (as Al<sub>2</sub> O<sub>3</sub>), percent by mass =  $\frac{M - M_1}{M_2} \times 100$ 

Where,

M = mass in g of the mixed oxides as obtained in B-1·3·7

M<sub>1</sub> = mass in g of the ferric oxide and titanium dioxide, together as calculated from B-2·4 and B-3·4 respectively.

 $M_2 = mass in g of the sample taken.$ 

#### B-5 DETERMINATION OF LOSS ON IGNITION

- B-5.1 Procedure Take about 50g of the material in a procelain basin and heat to constant weight at 105° ± 2°C. Cool in a desiccator.
  - B-5·1·1 Heat 1g of the above dried material in a platinum crucible for 15 minutes over a small flame, then for 30 minutes over a good Meker burner, with the lid on for the last 10 minutes. Cool the crucible and contents in a desiccator, weigh again, heat for 10 minutes over the Meker burner, and then weigh again after cooling. The loss in mass denotes loss on ignition.
- B-5.2 Calculation Calculate the loss on ignition as follows:
  Loss on ignition,

percent by mass = 100 x 
$$\frac{M_1 - M_2}{M_1}$$

where.

M<sub>1</sub> = mass in g of the dry material, and

M<sub>2</sub> = mass in g of the material after ignition.

## APPENDIX 'C'

(Clause 2 · 3 · 1)

## DETERMINATION OF MOISTURE C-1 PROCEDURE

C-1·1 Spread uniformly on to a dry, weighed petri dish about 1 to 2g of the thoroughly mixed clay sample (see 4·2·2) and weigh accurately. Dry it in an air oven at 105°C to 110°C for three hours, cool in a desiccator and weigh. Repeat the process till constant mass is obtained. Record the loss in mass of the sample.

#### C-1.2 Calculation

Moisture, percent = 
$$\frac{M_1}{M_2}$$
 x 100

where,

M<sub>1</sub> = loss of weight on drying in g, and

M<sub>2</sub> = weight in g of the sample taken.



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